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INFRARED SPECTROSCOPY

Unlike the chromatographies, which physically separate materials, **infrared (IR) spectroscopy** is a method of determining what you have after you've separated it.

The **IR spectrum** is the name given to a band of frequencies between 4000 and 650 cm^{-1} beyond the red end of the visible spectrum. The units are called **wave numbers** or **reciprocal centimeters** (that's what cm^{-1} means). This range is also expressed as wavelengths from 2.5 to 15 micrometers (μ).

With your sample in the **sample beam**, the instrument scans the IR spectrum. *Specific functional groups absorb specific energies*. Because the spectrum is laid out on a piece of paper, these specific energies become *specific places* on the chart.

Look at Fig. 149. Here's a fine example of a pair of alcohols if ever there was one. See the peak (some might call it a trough) at about 3400 cm^{-1} (2.9 μ)? That's due to the **OH group**, specifically the stretch in the O—H bond, the **OH stretch**.

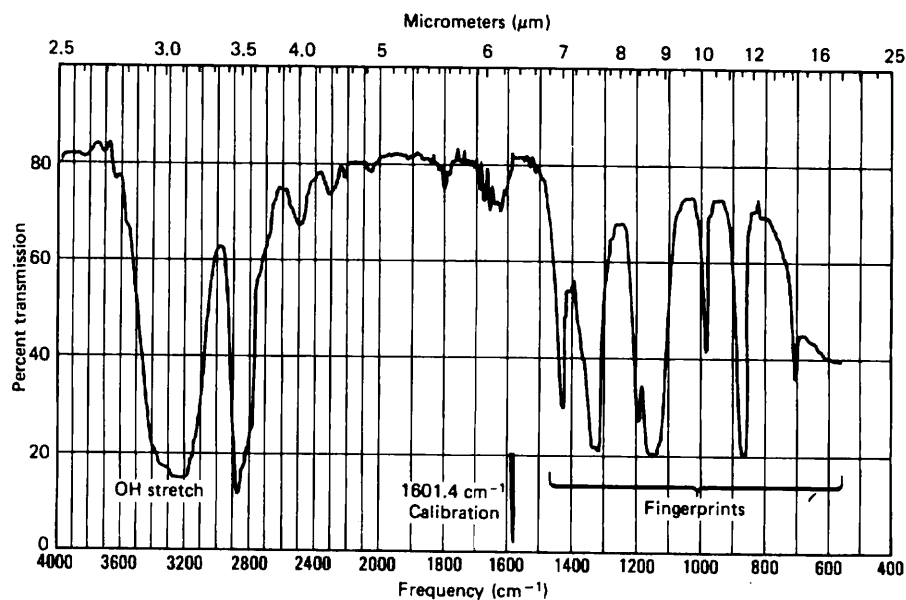
Now consider a couple of ketones, 2-butanone and cyclohexanone (Fig. 150). There's no OH peak about 3400 cm^{-1} (2.9 μ), is there? Should there be? *Of course not*. Is there an OH in 2-butanone? *Of course not*. But there is a C=O, and where's that? The peak about 1700 cm^{-1} (5.9 μ). It's *not there* for the alcohols, and it *is* there for the ketones. Right. You've just correlated or *interpreted* four IRs.

Because the first two (Fig. 149) have the *characteristic OH stretch of alcohols*, they might just be alcohols. The other two (Fig. 150) might be ketones because of the *characteristic C=O stretch* at 1700 cm^{-1} (5.9 μ) in each.

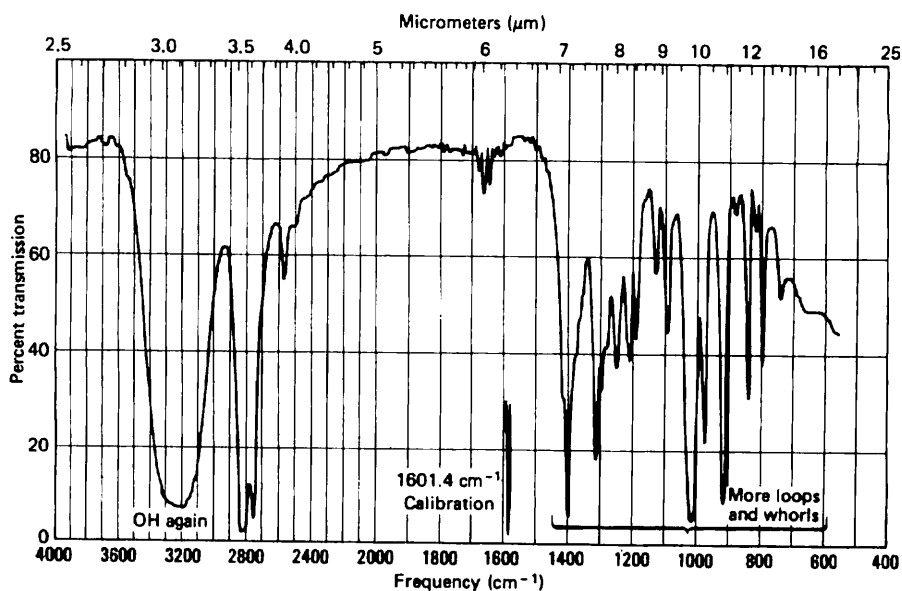
What about all the other peaks? You *can* ascribe some sort of meaning to each of them, but it can be very difficult. That's why **frequency correlation diagrams**, or **IR tables**, exist (Fig. 151). They identify regions of the IR spectrum where peaks for various functional groups show up. They can get very complicated. Check to see if you can find the C—H stretch and the C—O stretch that are in all four spectra, using the correlation table. It can be fun.

For you Sherlock Holmes fans, the region from 1400 to 990 cm^{-1} (7.2 to 11.1 μ) is known as the **fingerprint region**. The peaks are due to the entire molecule, *its fingerprint*, rather than being from independent functional groups. And, you guessed it, no two fingerprints are alike.

Take another look at the cyclohexanol and cyclohexanone spectra. Both have very different functional groups. Now look at the similarities, the simplicity, including the fingerprint region. Both are six-membered rings



(a)



(b)

Fig. 149 IR spectra of (a) t-butanol and (b) cyclohexanol.

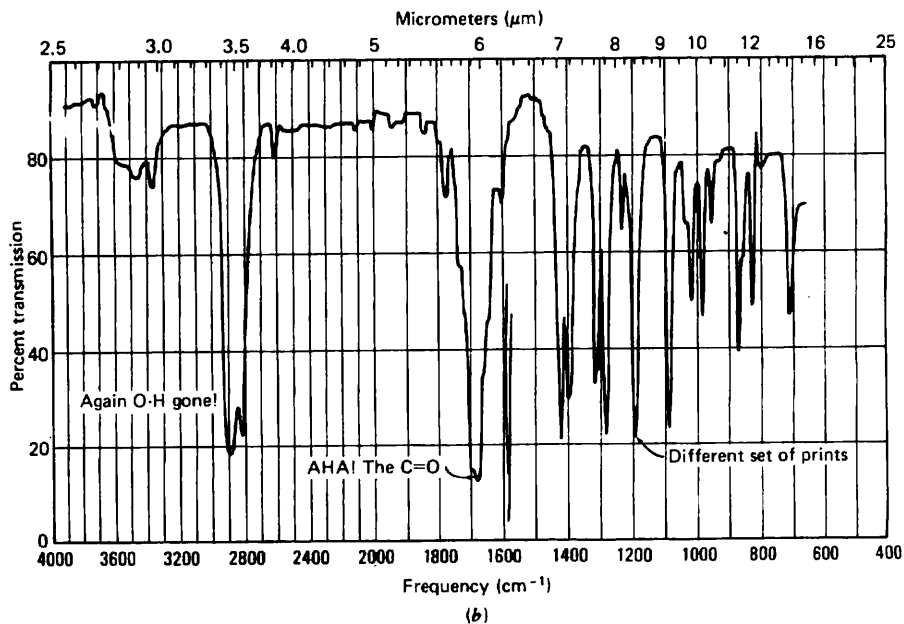
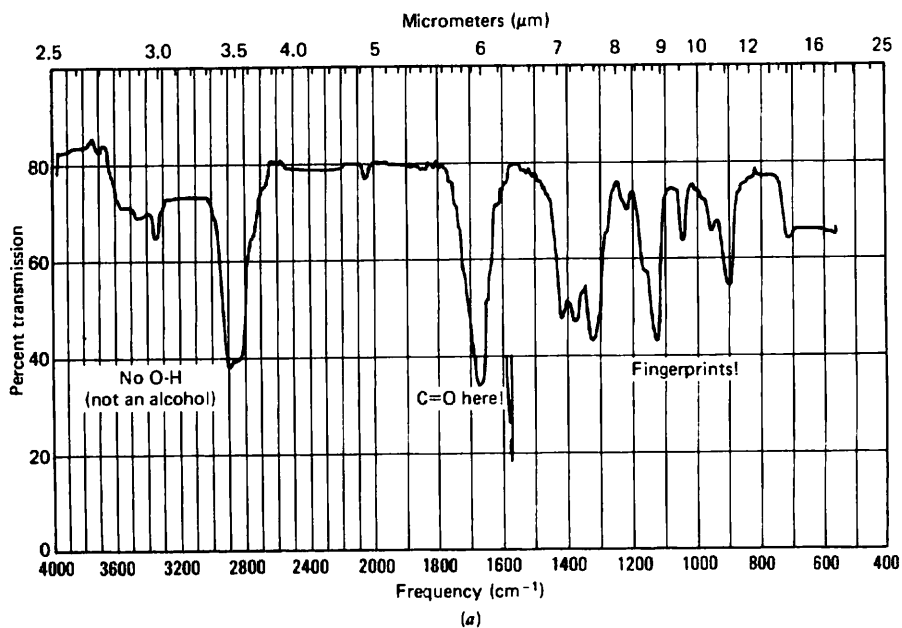


Fig. 150 IR spectra of (a) 2-butanone and (b) cyclohexanone.

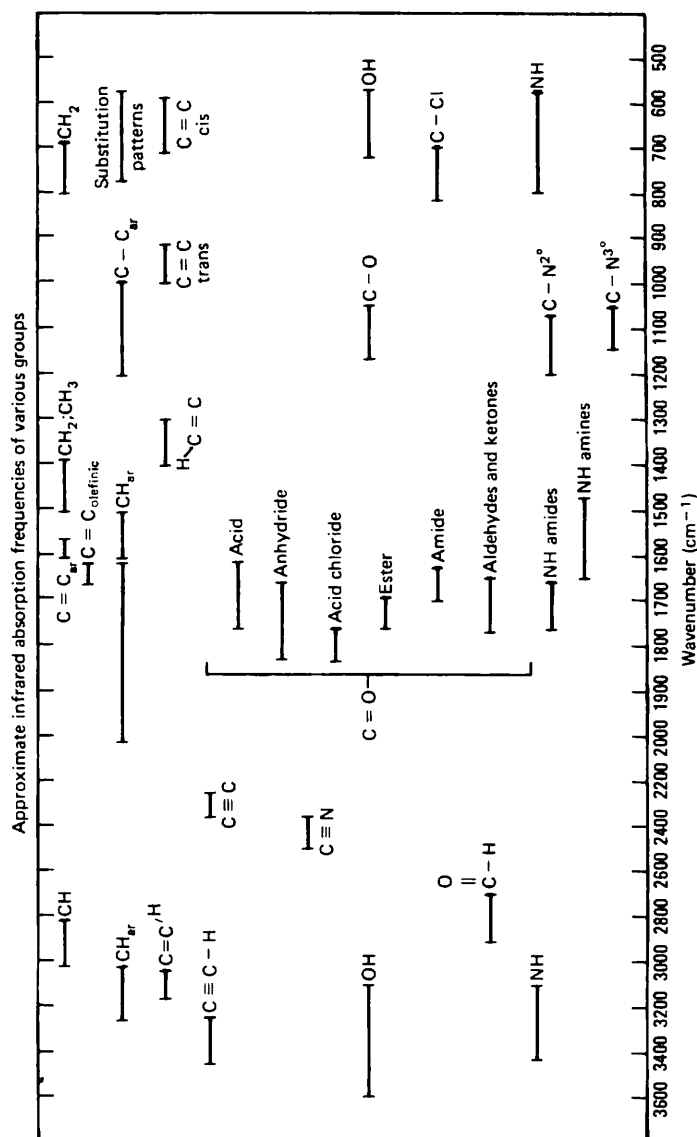


Fig. 151 The author's only IR correlation chart.

and have a high degree of symmetry. You should be able to see the similarities owing to the similar structural features.

Two more things. First, watch your spelling and pronunciation; it's not "infared," OK? Second, most people I know use IR (pronounced "eye-are," not "ear") to refer to the technique, the instrument, and the chart recording of the spectrum:

<i>"That's a nice new IR you have there."</i>	(the instrument)
<i>"Take an IR of your sample."</i>	(perform the technique)
<i>"Let's look at your IR and see what kind of compound you have."</i>	(interpret the resulting spectrogram)

To take an IR, you need an IR. These are fairly expensive instruments; again, no one is typical, but you can get a feeling of how to run an IR as you go on.

INFRARED SAMPLE PREPARATION

You can prepare samples for IR spectroscopy easily, but you must strictly adhere to one rule:

No water!

In case you didn't get that the first time:

No water!

Ordinarily, you put the sample between two salt plates. Yes. Common, ordinary *water-soluble* salt plates. Or mix it with **potassium bromide (KBr)**, another water-soluble salt.

So keep it dry, people.

Liquid Samples

1. Make sure the sample is DRY. NO WATER!
2. Put some of the *dry* sample (2–3 drops) on one plate, then cover it with another plate (Fig. 152). The sample should spread out to cover the entire plate. You *don't have to press*. If it doesn't cover well, try turning the top plate to spread the sample, or add more sample.
3. Place the sandwich in the IR salt plate holder and cover it with a hold-down plate.

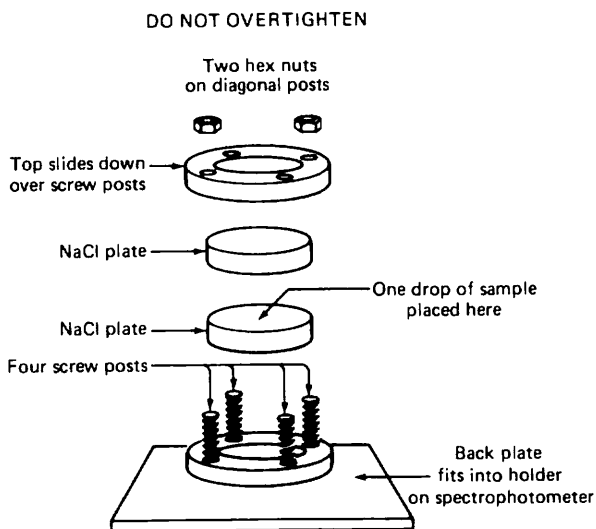


Fig. 152 IR salt plates and holder.

4. Put at least two nuts on the posts of the holder (opposite corners) and spin them down *GENTLY* to hold the plates with an even pressure. *Do not use force!* You'll crack the plates! Remember, these are called salt plate holders and not salt plate smashers.
5. Slide the holder and plate into the bracket in the sample beam (closer to you, facing the instrument.)
6. Run the spectrum.

Since you don't have any other solvents in there, just your liquid compound, you have just prepared a liquid sample, **neat**, meaning *no solvent*. This is the same as a *neat liquid sample*, which is a way of describing any liquid without a solvent in it. It is not to be confused with a "really neat liquid sample," which is a way of expressing your true feelings about your sample.

Solid Samples

The Nujol Mull

A rapid, inexpensive way to get an IR of solids is to mix them with Nujol, a commercially available mineral oil. Traditionally, this is called "making

a Nujol mull,” and it is practically idiomatic among chemists. Although you won’t see Rexall or Johnson & Johnson mulls, the generic brand **mineral oil mull** is often used.

You want to disperse the solid throughout the oil, making the solid transparent enough to IR that the sample will give a usable spectrum. Since mineral oil is a saturated hydrocarbon, it has an IR spectrum all its own. You’ll find hydrocarbon bends, stretches, and pushups in the spectrum, but you know where they are, and you ignore them. You can either look at a published reference Nujol spectrum (Fig. 153) or run your own if you’re not sure where to look.

1. Put a small amount of your solid into a tiny agate mortar and add a few drops of mineral oil.
2. Grind the oil and sample together until the solid is a fine powder *dispersed throughout the oil*.
3. Spread the mull on one salt plate and cover it with another plate. There should be no air bubbles, just an even film of the solid in the oil.
4. Proceed as if this were a *liquid sample*.
5. Clean the plates with *anhydrous* acetone or ethanol. **NOT WATER!** If you don’t have the tiny agate mortar and pestle, try a Witt spot plate and the rounded end of a thick glass rod. The spot plate is a piece of glazed porcelain with dimples in it. Use one as a tiny mortar and the other as a tiny pestle.

And remember to forget the peaks from the Nujol itself.

Solid KBr Methods

KBr methods (hardly ever called potassium bromide methods) consist of making a mixture of your solid (*dry again*) with IR-quality KBr. Regular KBr off the shelf is likely to contain enough nitrate, as KNO_3 , to give spurious peaks, so don’t use it. After you have opened a container of KBr, dry it and later store it in an oven, with the cap off, at about 110°C to keep the moisture out.

Preparing the Solid Solution

1. At least once in your life, *weigh out* 100 mg of KBr so you’ll know how much that is. If you can remember what 100 mg of KBr looks like, you won’t have to weigh it out every time you need it for IR.

CONCENTRATION _____	SPECTRUM NO. _____ SAMPLE _____ ORIGIN _____
THICKNESS _____	
PHASE _____	
REMARKS _____	
OPERATOR _____ DATE _____	

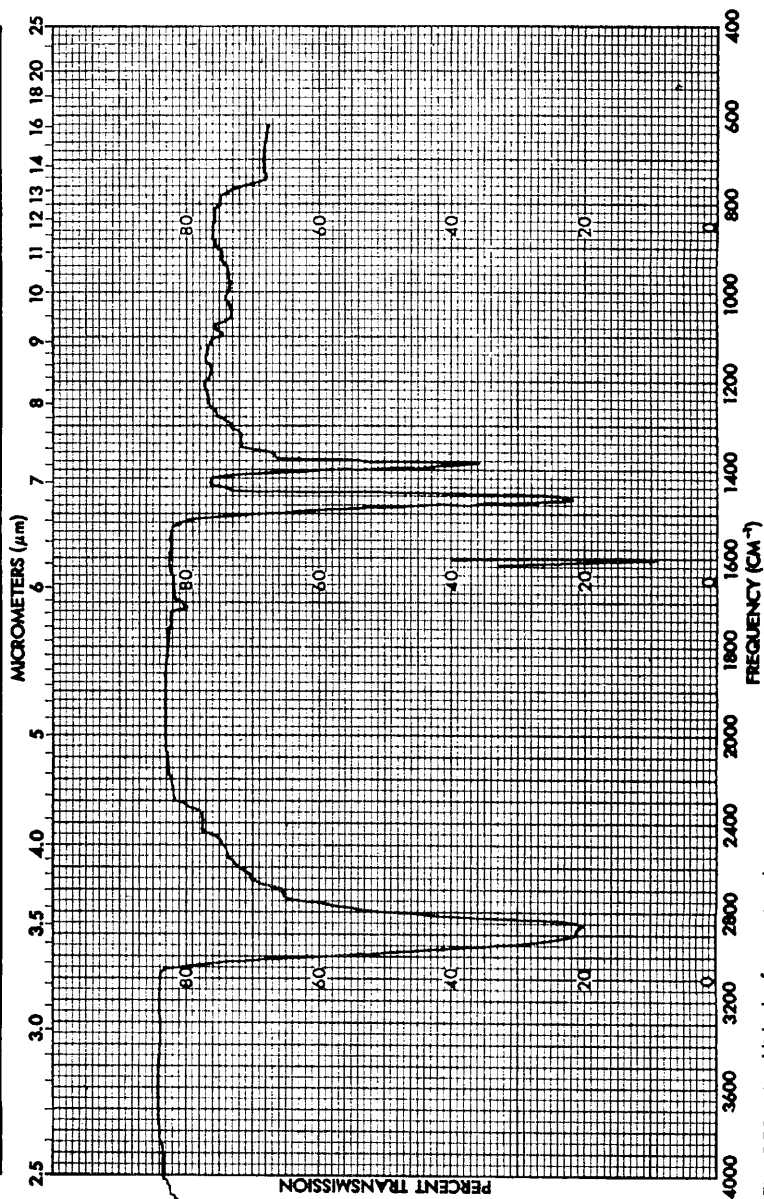


Fig. 153 A published reference Nujol spectrum.

2. Weigh out 1–2 mg of your *dry, solid* sample. You'll have to weigh out each *sample* because different compounds take up different amounts of space.
3. Pregrind the KBr to a fine powder about the consistency of powdered sugar. Don't take forever, since moisture from the air will be coming in.

Pressing a KBr Disk—The Mini-Press (Fig. 154)

1. Get a clean, dry press and two bolts. Screw one of the bolts into the press about halfway and call that the bottom of the press.
2. Scrape a finely ground mixture of your compound (1–2 mg) and KBr (approximately 100 mg) into the press so that an even layer covers the bottom bolt.
3. Take the other bolt and turn it in from the top. *Gently* tighten and loosen this bolt at least once to spread the powder evenly on the face of the bottom bolt.
4. Hand tighten the press again, and then use wrenches to tighten the bolts against each other. Don't use so much force that you turn the heads right off the bolts.
5. Remove both bolts. A KBr pellet, containing your sample, should be in the press. Transparent is excellent. Translucent will work. If the sample is opaque, you can run the IR, but I don't have much hope of your finding anything.
6. Put the entire press in a holder placed in the analyzing beam of the IR, as in Fig. 155. (Don't worry about that yet; I'll get to it in a moment. See "Running the Spectrum" in the next major section.)

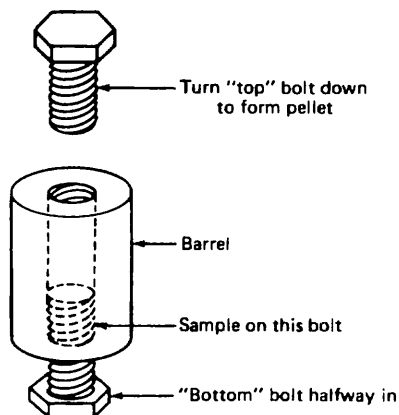


Fig. 154 The mini-press.

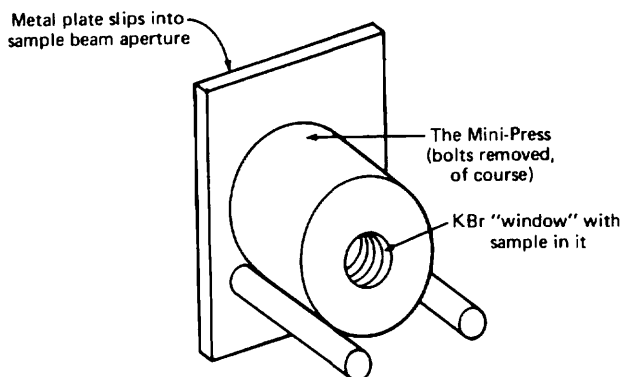


Fig. 155 The mini-press in its holder.

Pressing a KBr Disk—The Hydraulic Press

If you have a hydraulic press and two steel blocks available, there is another easy KBr method. It's a card trick, and at no time do my fingers leave my hands. The only real trick is you'll have to bring the card.

1. Cut and trim an index card so that it fits into the **sample beam aperture** (Fig. 160).
2. Punch a hole in the card with a paper punch. The hole should be *centered in the sample beam* when the card is in the sample beam aperture.
3. Place one of the two steel blocks on the bottom jaw of the press.
4. Put the card on the block.
5. Scrape your KBr-sample mixture onto the card, covering the hole and some of the card. Spread it out evenly.
6. Cover the card, which now has your sample on it, with the second metal block (Fig. 156).
7. Pump up the press to the indicated *safe* pressure.
8. Let this sit for a bit (1 minute). If the pressure has dropped, bring it back up, *slowly, carefully*, to the safe pressure line, and wait another bit (1 minute).
9. Release the pressure on the press. Push the jaws apart.
10. Open the metal sandwich. Inside will be a file card with a **KBr window** in it, just like in the Mini-Press.

CAUTION! *The KBr window you form is rather fragile, so don't beat on it.*

11. Put the card in the **sample beam aperture** (Fig. 157). The KBr window should be *centered* in the sample beam if you've cut and punched the card correctly.

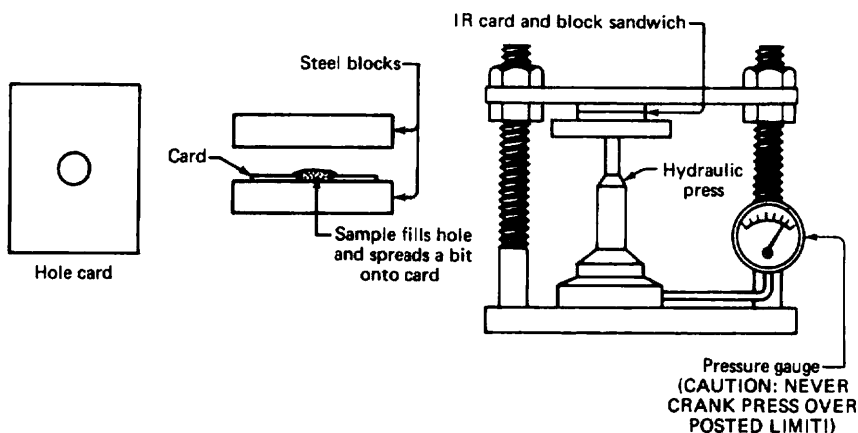


Fig. 156 KBr disks by hydraulic press.

RUNNING THE SPECTRUM

There are many IR instruments, and since they are so different, you need your instructor's help here more than ever. But there are a few things you have to know.

1. **The sample beam.** Most IRs are **dual-beam instruments** (Fig. 158). The one closest to you, if you're operating the instrument, is the **sample beam**. Logically, there is a **sample holder** for the sample beam, and your sample goes there. And there, a beam of IR radiation goes through your sample.
2. **The reference beam.** This is the other light path. It's not *visible* light but another part of the electromagnetic spectrum. Just remember that the reference beam is the one farthest away from you.
3. **The 100% control.** This sets the pen at the 100% line on the chart paper. Or tries to. It's a very delicate control and doesn't take kindly to excessive force. Read on, and all will be made clear.
4. **The pen.** There is a pen and pen holder assembly on the instrument. This is how the spectrum gets recorded on the chart paper. Many people get the urge to throw the instrument out the window when the pen stops writing in the middle of the spectrum. Or doesn't even start writing. Or was left empty by the last fellow. Or was left to dry out on the top of the instrument. For those with Perkin-Elmer 137s and 710s, two clever fellows have made up generic felt-tip pen holders for the instruments. This way, you buy your own pen from the bookstore, and

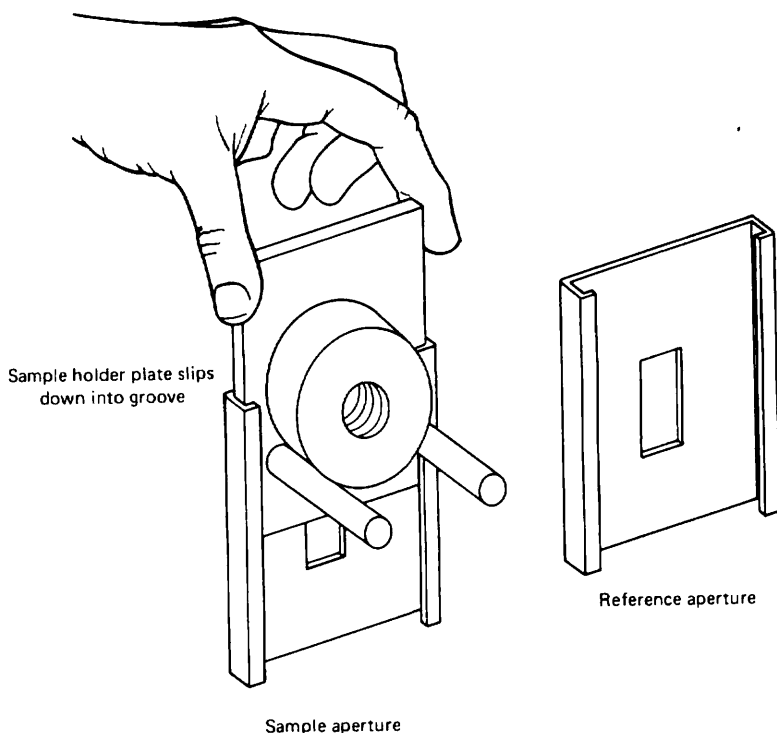


Fig. 157 Putting sample holders with samples into the beam.

if it dries out it's your own fault. [See R. A. Bailey and J. W. Zubrick, *J. Chem. Educ.*, 59, 21 (1982).]

5. **The very fast or manual scan.** To get a good IR, you'll have to be able to scan, very rapidly, *without letting the pen write on the paper*. This is so you'll be able to make adjustments before you commit pen to paper. This **fast forward** is not a standard thing. Sometimes you operate the instrument by hand, pushing or rotating the paper holder. Again, *do not use force*.

Whatever you do, *don't try to move the paper carrier by hand when the instrument is scanning a spectrum*. Stripped gears is a crude approximation of what happens. So, thumbs off.

I'm going to apply these things in the next section using a real instrument, the Perkin-Elmer 710B, as a model. Just because you have another model is no reason to skip this section. If you do have a different IR, try to find the similarities between it and the Perkin-Elmer model. Ask your instructor to explain any differences.

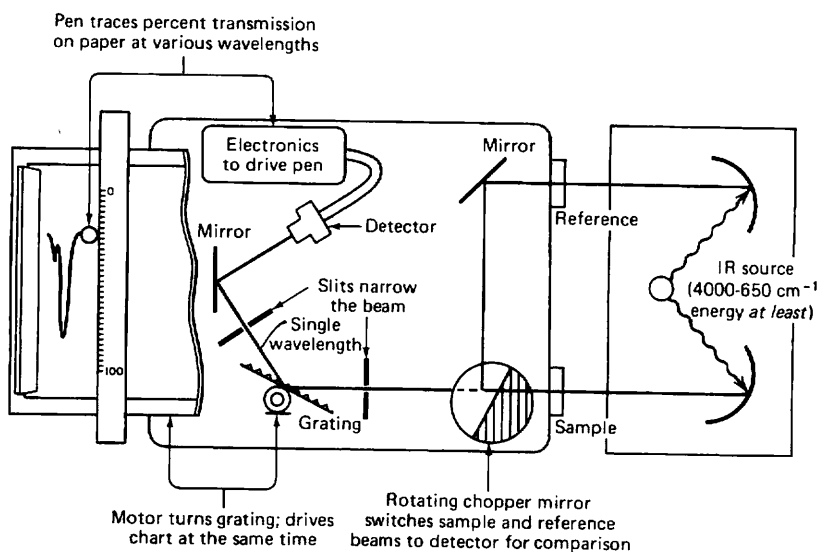


Fig. 158 Schematic diagram of an IR.

THE PERKIN-ELMER 710B IR (FIG. 159)

1. **On-off switch and indicator.** Press this once, the instrument comes on, and the switch lights up. Press this again, the instrument goes off, and the light goes on.
2. **Speed selector.** Selects speed (normal or fast). "Fast" is faster, but slower gives *higher resolution*, that is, more detail.
3. **Scan control.** Press this to *start a scan*. When the instrument is scanning, the optics and paper carrier move automatically, causing the IR spectrum to be drawn on the *chart paper* by the *pen*.
4. **Chart paper carriage.** This is where the chart paper nestles while you run an IR. If it looks suspiciously like a clipboard, it's because that's how it works.
5. **Chart paper hold-down clip.** Just like a clipboard, this holds the paper down in the carrier.
6. **Frequency scale.** This scale is used to help align the chart paper and to tell you during the run where in the spectrum the instrument is.
7. **Scan position indicator.** A white arrow that points, roughly, to where the instrument is in the spectrum.

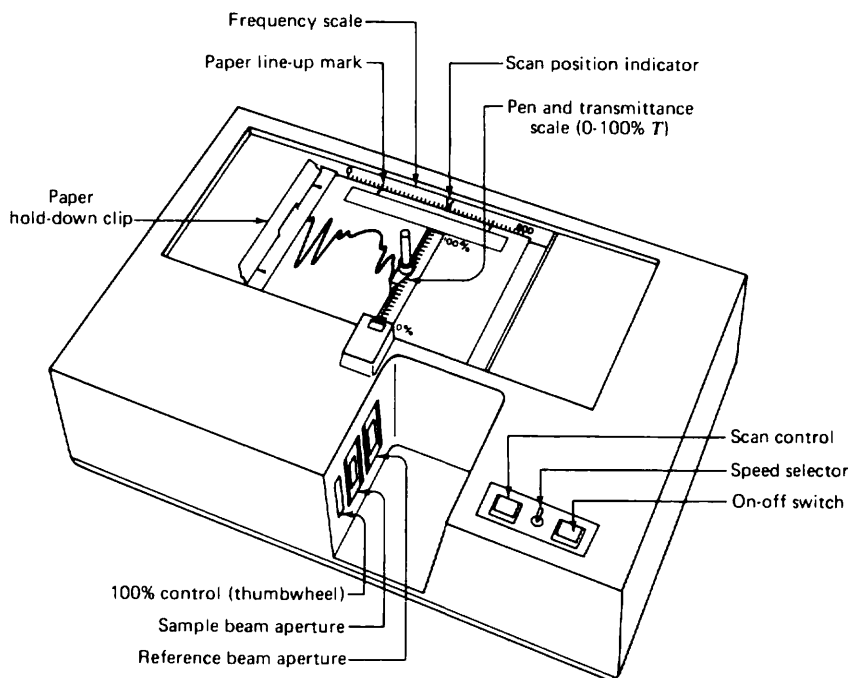


Fig. 159 The Perkin-Elmer 710B IR.

8. **Line-up mark.** A line, here at the number 4000, that you use to match up the numbers on the instrument frequency scale with the same numbers on the chart paper.
9. **Pen and transmittance scale.** This is where the pen traces your IR spectrum. The numbers here mean percentage of IR transmitted through your sample. If you have no sample in the sample beam, how much of the light is getting through? Those who said 100% are 100% correct. Block the beam with your hand and 0% gets through. You should be able to see why these figures are called the % **transmission**, or %T, scale.
10. **The 100% control.** Sets the pen at 100%. Or tries to. This is a fairly sensitive control, so don't force it.
11. **Sample beam aperture.** This is where you put the holder containing your sample, be it mull or KBr pellet. You slip the holder into the aperture window for analysis.
12. **Reference beam aperture.** This is where nothing goes. Or, in extreme cases, you use a **reference beam attenuator** to cut down the amount of light reaching the detector.

USING THE PERKIN-ELMER 710B

1. Turn the instrument on and let it warm up for about 3–5 minutes. Other instruments may take longer.
2. Get a piece of IR paper and load the *chart paper carriage*, just like a clipboard. Move the paper to get the index line on the paper to line up with the index line on the instrument. It's at 4000cm^{-1} and it's only a rough guide. Later I'll tell you how to calibrate your chart paper.
3. Make sure the chart paper carriage is at the high end of the spectrum 4000cm^{-1} .
4. Put your sample in the sample beam. Slide the sample holder with your sample into the sample beam aperture (Fig. 157).
5. What to do next varies for particular cases—not much but enough to be confusing in setting things up.
6. Look at where the pen is. *Carefully* use the **100% control** to locate the pen at about the 90% mark when the chart (and spectrum) is at the high end 4000cm^{-1} .

The 100% Control: An Important Aside

Usually, there's not much more to adjusting the 100% control than is shown in upcoming items 7 through 9, unless your sample, *by its size alone*, reduces the amount of IR reaching the detector. This really shows up if you've used the mini-press, which has a *much smaller opening* than that of the opening in the reference beam. So you're at a disadvantage right from the start. Sometimes the 100% control mopes around at much less than 40%. That's terrible, and you'll have to use a **reference beam attenuator** (Fig. 160) to equalize the amounts of energy in the two beams. As you block more and more of the energy in the reference beam, the %*T* will go back to the 100% mark. Stop at about 90%. Note that, if you didn't have these problems, this is where you'd put the pen with the 100% control anyway. Use the *smallest amount* of reference beam attenuation you can get away with.

7. OK, at 4000cm^{-1} the %*T* (the pen) is at 90%.
8. Now *slowly, carefully*, move the pen carriage manually so that the instrument scans the entire spectrum. *Watch the pen!* If the **baseline**

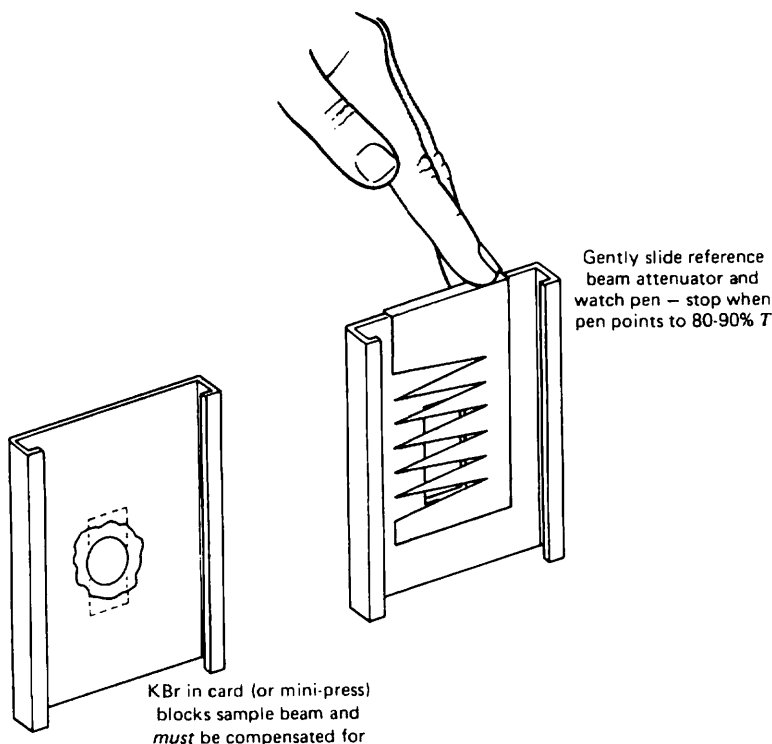


Fig. 160 Using a reference beam attenuator with a KBr window.

creeps up and goes off the paper (Fig. 161), this is not good. Readjust the 100% control to keep the pen on the paper. Now keep going, slowly, and *if* the pen drifts up again, readjust it again with the 100% control and get the pen back on the paper.

9. Now go back to the beginning (4000 cm^{-1}). If you've adjusted the 100% control to get the pen back on the paper at some other part of the spectrum, surprise! The pen will not be at 90% when you get back. This is unimportant. What is important is that *the pen stay within the limits, between the 0 and 100%T lines, for the entire spectrum.*
10. In *any* case, if the peaks are too large, with the baseline in the proper place, your sample is just *too concentrated*. You can wipe some of your liquid sample or mull off one of the salt plates or remake the KBr pellet using less compound or more KBr. Sorry.
11. When you've made all the adjustments, press the scan button and you're off.

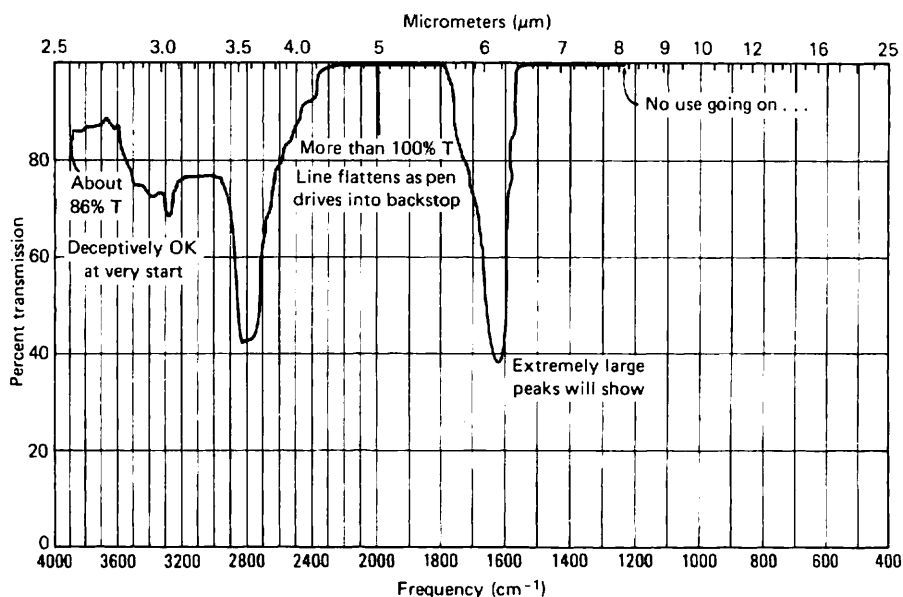


Fig. 161 An IR with an unruly baseline.

CALIBRATION OF THE SPECTRUM

Once the run is over, there's one other thing to do. Remember that the index mark on the paper is not exact. You have to **calibrate the paper** with a standard, usually *polystyrene film*. Some of the peaks in polystyrene are quite sharp, and many of them are very well characterized. A popular one is an extremely narrow, very sharp spike at 1601.4 cm^{-1} (6.24μ).

1. *Don't move the chart paper*, or this calibration will be worthless.
2. Remove your sample, and replace it with the standard polystyrene film sample. You will have to remove any reference beam attenuator and turn the 100% control to set the pen at about 90%, when the chart is at 4000 cm^{-1} .
3. *With the pen off the paper*, move the carriage so that it's *just before* the calibration peak you want, in this case 6.24μ .
4. Now, quickly, start the scan, *let the pen draw just the tip of the calibration peak*, and quickly stop the scan. You don't need to draw more than that (Fig. 162). Just make sure you can pick your calibration peak out of the spectral peaks. If it's too crowded at 1601.4 cm^{-1} , use a different polystyrene peak— 2850.7 or 1028.0 cm^{-1} .

(3.51 or 9.73 μ). Anything really well known and fairly sharp will do (Fig. 163).

5. And that's it. You have a nice spectrum.

32

IR SPECTRA: THE FINISHING TOUCHES (FIG. 164)

IR chart paper contains spaces for all sorts of information. It would be nice if you could fill in

1. **Operator.** The person who ran the spectrum. Usually you.
2. **Sample.** The name of the compound you've just run.
3. **Date.** The day you ran the sample.
4. **Phase.** For KBr, say "solid KBr." A Nujol mull is "Nujol mull." Liquids are either solutions in solvents or "neat liquids," that is, without any solvents, so call them liquids.
5. **Concentration.** For KBr, a solid solution, list milligrams of sample in 100 mg of KBr. For liquids, *neat* is used for liquids without solvents.

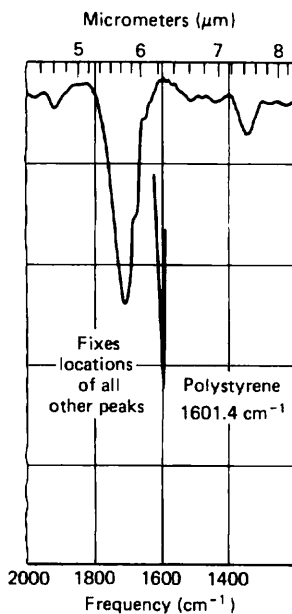


Fig. 162 A calibration peak on an IR spectrum.

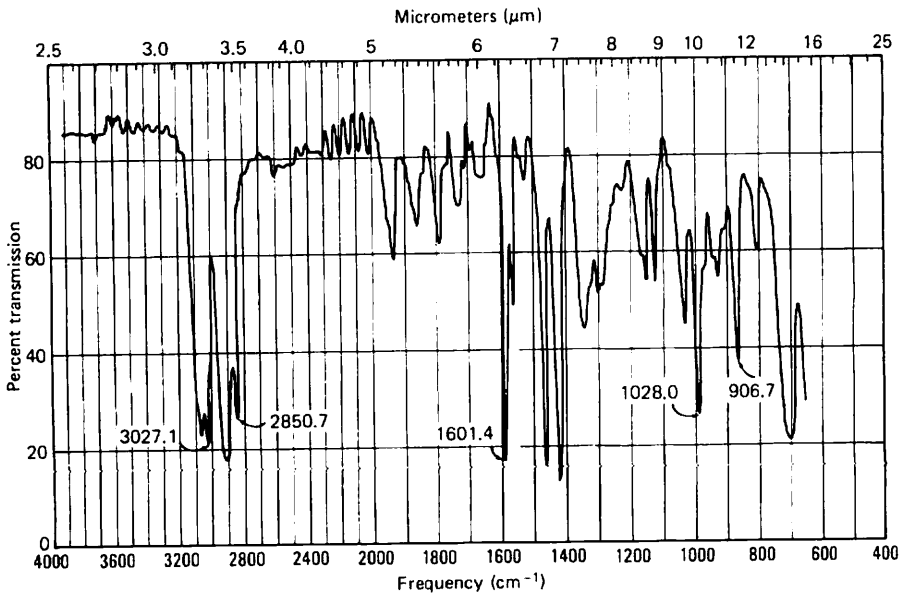


Fig. 163 IR of polystyrene film pointing out many calibration peaks.

CONCENTRATION <u>Neat</u>		SPECTRUM NO. <u>6</u>
THICKNESS <u>Thin film</u>		SAMPLE <u>Cyclohexanone</u>
PHASE <u>Liquid</u>		ORIGIN <u>Student prep.</u>
REMARKS <u>1601.4 cm⁻¹ Calibration</u>		OPERATOR <u>J. W. Zurcher</u> DATE <u>11/30/82</u>

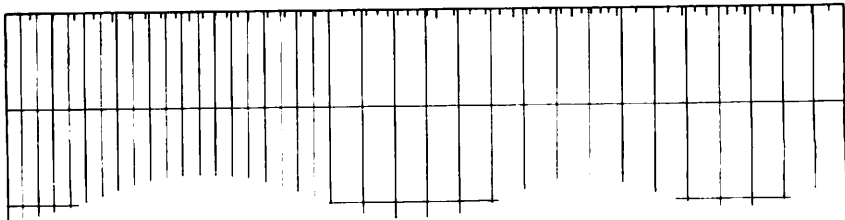


Fig. 164 The finishing touches on the IR.

6. **Thickness.** Unless you're using solution cells, write **thin film** for **neat liquids**. Leave this blank for KBr samples (unless you've measured the thickness of the KBr pellet, which you shouldn't have done).
7. **Remarks.** Tell where you put your calibration peak, where the sample came from, and anything unusual that someone in another lab might have trouble with when trying to duplicate your work. Don't put this off until the last day of the semester when you can no longer remember the details. Keep a record of the spectrum *in your notebook*.

You now have a perfect IR, suitable for framing and interpreting.

INTERPRETING IR's

IR interpretation can be as simple or as complicated as you'd like to make it. You've already seen how to distinguish alcohols from ketones by **correlation** of the positions and intensities of various peaks in your spectrum with positions listed in **IR tables** or **correlation tables**. This is a fairly standard procedure and is probably covered very well in your textbook. The things that are not in your text are as follows.

1. **Not forgetting the Nujol peaks.** Mineral oil will give huge absorptions from all the C—H bonds. They'll be the biggest peaks in the spectrum. And every so often, people mistake one of these for something that belongs with the sample.
2. **Nitpicking a spectrum.** Don't try to interpret every wiggle. There is a lot of information in an IR, but sometimes it is confusing. Think about what you're trying to show and, then show it.
3. **Negative results,** Negative results can be very useful. No big peak between 1600 and 2000 cm^{-1} ? No carbonyl. Period. No exceptions.
4. **Pigheadedness in interpretation.** Usually a case of, "I know what this peak is so don't confuse me with facts." Infrared is an extremely powerful technique, but there are limitations. You don't have to go hog wild over your IR, though. I know of someone who decided that a small peak was an N-H stretch, and the compound *had* to have nitrogen in it. The facts that the intensity and position of the peak were not quite right, and neither a chemical test nor solubility studies indicated nitrogen, didn't matter. Oh well.

THE FOURIER TRANSFORM INFRA-RED (FTIR)

There's even more of a difference in particulars between FTIRs than between dispersive IRs (Like the 710B mentioned earlier), because not only can the basic instrument vary, but it can be attached to any number of different computers of differing configurations running any number of different software programs doing the infra-red analysis. Phew. So I'll only hit the major differences.

The Optical System

The optical system is based upon the Michelson Interferometer (Fig. 165). Infra-red energy from the source goes through the sample in a single beam and hits a beamsplitter in the interferometer. Half of the light gets reflected to a stationary mirror, and the other half passes through the beamsplitter to a moving mirror. Both mirrors reflect the light back to the beamsplitter, where they recombine to form an interference pattern of constructive and destructive interferences known as **interferogram**. (Consider the pattern produced when sets of waves from two stones thrown into a pond interact: The peak of one and the peak of the other re-inforce each other, giving a bigger peak; the peak of one and the trough of the other can completely destroy each other and produce no displacement.) That interference pat-

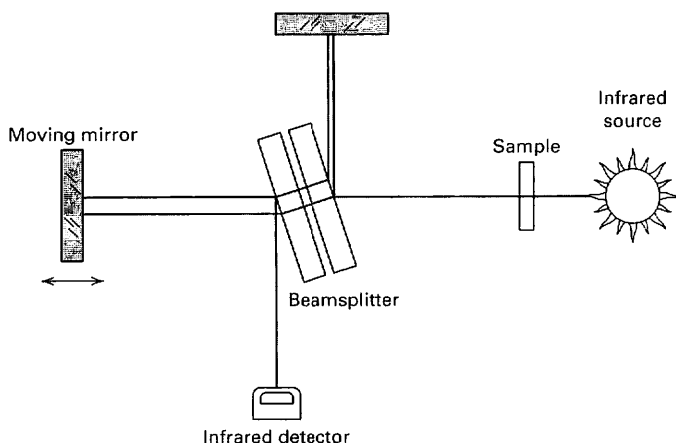


Fig. 165 Michelson Interferometer Optical System for FTIR.

tern varies with the displacement of the moving mirror, and this pattern of variation is sent along to the detector.

A computer programmed with the algorithm of the Fourier transformation converts the measured-intensity-versus-mirror-displacement signal (the interferogram) into a plot of intensity versus frequency—our friendly infra-red spectrum.

FTIR has a few advantages:

1. ***Fellgett's Advantage (Multiplex Advantage)***. The interferometer doesn't separate light into individual frequencies like a prism or grating, so every point in the interferogram (spectrum) contains information from each wavelength of infra-red from the source.
2. ***Jacquinot's Advantage (Throughput Advantage)***. The dispersive instrument, with prism or grating, needs slits and other optics so as much of a single wavelength of energy reaches the detector as possible. Not so the interferometer. So the entire energy of the source comes roaring through the optical system of the interferometer to the detector, increasing the signal-to-noise ratio of the spectrum.
3. ***Conne's Advantage (Frequency Precision)***. The dispersive instrument depends on calibration (polystyrene at 1601 cm^{-1}), and the ability of gears and levers to move slits and gratings in a reproducible fashion. The FTIR carries its own internal frequency standard, usually a He-Ne gas laser, that serves as the master timing clock, tracking mirror movement and frequency calibration to a precision and accuracy of better than $0.01\text{ wavenumbers (cm}^{-1}\text{)}$.

But what about your advantages. And your disadvantages?

1. ***FTIR is a single-beam instrument***. So you must collect a background spectrum (Figure 166) before you do your sample. The computer program will subtract the background from the background-and-sample and produce the usual IR spectrum. What's in a background spectrum? Everything that's not the sample. For a KBr disk spectrum, the empty press (Figure 155) or an empty card (Figure 156) gets included in the background spectrum. For a salt plate spectrum (Figure 152), clean salt plates in the holder is the background. And for a solution-cell spectrum, the cell with pure solvent is the background. Why? For many cells and holder combinations, the aperture is smaller, or the salt plates absorb a bit, or the solvent absorbs a lot, and these must be compensated for.

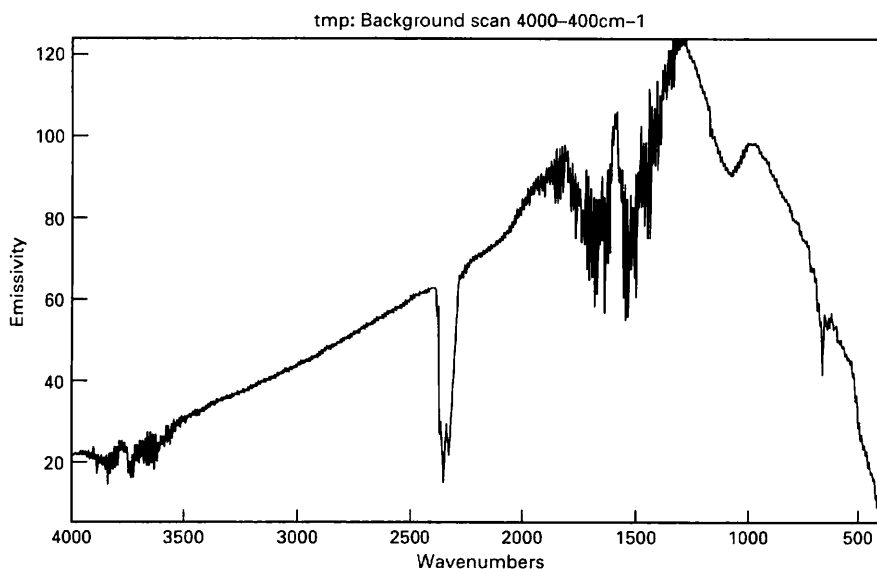


Fig. 166 FTIR background spectrum. Note H_2O and CO_2 bands.

2. **The FTIR is fast.** I can get the spectrum of a compound on my computer screen in about 0.21 minutes; considerably faster than any disperse instrument. Unfortunately, our inexpensive plotter is a bit slow, and, as the software makes the plotter label the axes with numbers and text, it does take a bit more than that 0.21 minutes. Overall, though, it is really considerably faster.
3. **The FTIR is accurate.** So you don't have to run a reference polystyrene. Unfortunately, we plot the spectra on inexpensive copier paper and if you don't mark relevant peaks on the screen with the software, you may never get those really accurate frequencies to 0.01 cm^{-1} .
4. **The FTIR computer programs automatically scale the spectrum.** So you don't have to set the zero and 100%. Unfortunately, the scaling is mindless, and you can be fooled if you're not careful. Figure 167 could possibly be an organic acid. See the broad hydrogen bonded OH peak at about 3400 cm^{-1} (The consequences of not marking the exact frequency with the computer program should be evident now.) But the $\text{C}=\text{O}$ at 1750 cm^{-1} isn't as strong as it should be and Fooled ya! Look at the transmittance scale on the left hand side. It only goes from 70 to 76%. This is a spectrum of some student-abused salt plates and these huge peaks and bands are actually tiny wiggles (Figure 168.). The computer program mindlessly took the maximum and minimum wiggling in the spectrum and decided to give it to you at full-screen height.

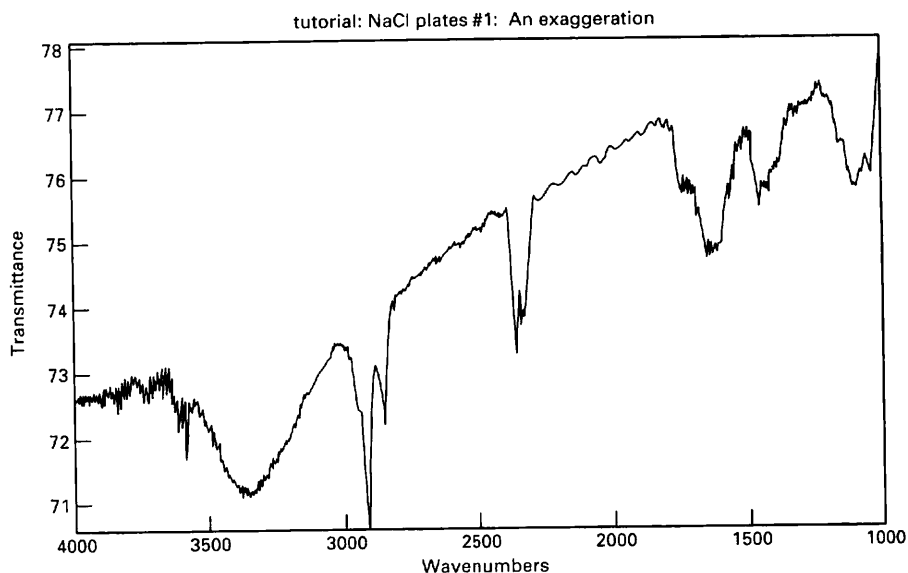


Fig. 167 Salt plate spectrum transmittance trick.

Admittedly, this is an unusual case. Since my background spectrum was taken with an empty salt-plate holder in the beam, my salt plates became the spectrum. Had I run the background with the holder and the cells, any bumps from the salt plates would have been cancelled.

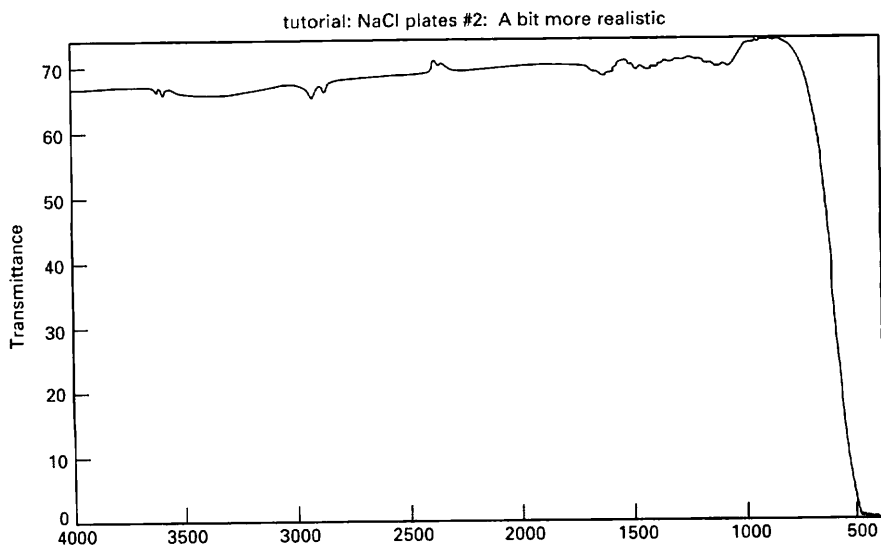


Fig. 168 Salt plate spectrum with some sanity restored.

And to get that 0 to 75% transmittance spectrum, I had to go outside the usual operating range of our instrument. Normally, you won't have such problems. But if you are now on your guard, you won't have *these* problems.

5. **The FTIR programs usually have a library spectral search, so you can easily identify your compounds.** Unfortunately, not every sample you'll run is in the database. So you could wind up with a collection of compounds usually having the same functional groups (and that can be helpful). Close—but no cigar. In that case, you must not succumb to the “I got it off the computer it must be right” syndrome.